



Exploration of soluble dietary fiber extraction technique for enhancing physicochemical and structural properties of mango and pomegranate peel

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Abstract

Fruit peel is the major bio-waste with no commercial value and possible industrial application. Different methods have already been employed for extraction of soluble dietary fiber (SDF) from various fruit peels. However, a sustainable and well reproducible method with efficient SDF yield is yet to uncover. Therefore, the present study attempted to decipher a new extraction method encompassed chemo-mechanical and thermal treatments among two varieties of Indian mango, i.e., totapuri and safeda (M1 and M2, respectively), and pomegranate, i.e., bhagwa and daru (P1 and P2, respectively), peel. The M1 sample resulted in maximum SDF yield as $44.35 \pm 0.66\%$ using a modified method employing chemical (vinegar 6%, and sodium hydroxide 0.4%), mechanical (ultrasonication at 150 W), and thermal (autoclaving at 121 °C/5 min) treatments. The spectroscopic and analytical characterization of SDF samples exhibited efficient structural properties with enhanced physicochemical characteristics. Therefore, the present study highlights the potential of a novel extraction method for the valorization of waste to a value-added functional food ingredient.

Keywords Soluble dietary fiber · Fruit peel · Waste/by-products · Chemo-mechanical and thermal treatment · Functional property

1 Introduction

India's production and processing of tropical fruits, namely, mango, banana, papaya, and pomegranate, have grown impressively in the past few years [1]. India positions second in the world's ranking of fruits and vegetables. However, the processing of these fruits generates a large number of by-products producing 5.6 million metric tons of annual waste creating serious environmental issues [2]. Among all processed fruits, mango and pomegranate generate almost 15–20% and 40–50% of their waste as peel respectively with no commercial value and possible industrial application [3, 4]. The processing of these fruits (to produce juice, puree,

and jams) generates enormous waste either in form of peel or pomace. Thus, the waste finds its use in animal feed or discarded simply as waste, owing to deficiency of processing facility or proper handling. Consequently, a substantial amount of nutrients is either lost or remained untapped [5–7]. Therefore, it is essential to utilize this waste to resolve the environmental problem and nutrient loss to boost the economy by increasing its industrial application. These residues are a good source of various bioactive components especially dietary fiber (DF) exhibiting health beneficiary effects [4]. Thus, the peel can serve as a bioactive ingredient and a potential source of DF.

DF is recognized as the 7th fundamental nutrient in an organism exhibiting a health beneficiary effect that includes facilitating intestinal transit and prevention against cancer, asthma, diabetes, and cardiovascular diseases [8, 9]. Based on its capability to form a solution in water, DF is generally classified as soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) [10]. SDF is more preferred due to its efficient solubility, oil/water holding capacity, interfacial and surface properties, and its potential of binding various molecules; comparatively, IDF is known to aid defecation

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along with bulking of stool [11, 12]. The proportion of SDF to IDF in the plant cell wall is naturally low; therefore, more efforts are required to find an appropriate and industrially feasible way to convert the maximum amount of IDF to SDF.

Currently, various conventional and non-conventional methods namely chemical, enzymatic, chemical-enzymatic, homogenization, ultrasonication, and microwave-assisted techniques have been employed for the extraction of SDF from different sources. However, less attention has been made to explore the effect of autoclaving treatment on the extraction of DF from agri-food waste/by-products. Though, to the best of our knowledge, there are no reports on the combined effect of acid, alkali, autoclaving, and ultrasonic treatments on the extraction of SDF from fruit peel as well as other fiber sources. Thus, in the present study, acid–alkali treatment combined with autoclaving and ultrasonication was employed to extract the SDF from two varieties of mango (totapuri and safeda) and pomegranate (bhagwa and daru) peel. The SDF extraction was carried out at different concentrations and samples with maximum SDF yield were further evaluated for physicochemical and functional properties along with structural characteristics. Overall, the present study aimed to decipher a novel, sustainable, and reproducible extraction method having possible feasibility at an industrial scale with the maximum conversion of IDF to SDF and to valorize waste into a value-added functional food ingredient.

2 Materials and methods

2.1 Material

The different varieties of mango, i.e., totapuri (M1) and safeda (M2), and pomegranate, i.e., bhagwa (P1) and daru (P2), were procured from the Palampur market (1472 m), Himachal Pradesh, India (32.1109° N, 76.5363° E). The total dietary fiber kit (K-TDFR, 200A) was procured from megazyme (Wicklow, Ireland). All standards were provided by Sigma-Aldrich, India. All other reagents and chemicals used to perform experiments were of HPLC and analytic grade.

2.2 Peel dietary fiber extraction

2.2.1 Pretreatment of peel powder

The fruits were thoroughly cleaned with running tap water and then peeled. Furthermore, the peel was dried in a hot air oven (Macro Scientific Work, Pvt, Ltd. India) for 48 h and ground into fine powder to get M1R (totapuri raw dietary fiber), M2R (safeda raw dietary fiber), P1R (bhagwa raw dietary fiber), and P2R (daru raw dietary fiber). Thereby, it was sieved using a 60-mm mesh size sieve. The powder was first treated with petroleum ether (10% w/v, 2 h) for the removal of various colors and lipids [13]. The solid residue was then further dried

at 40 °C and immersed in 70% ethanol (overnight) at 10% w/v for removal of organic acids, sugars of lower-molecular weight, inorganic salts, and inactivation of various enzymes [14].

2.2.2 SDF extraction from pretreated peel

The pretreated peel powder at concentrations 2%, 5%, and 10% (w/v) was dispersed in vinegar (6% glacial acetic acid; pH 2.14), and then autoclaved at 121 °C for 15 min. The supernatant was collected and the residue was washed three times with distilled water (DW). Furthermore, the residue was treated with 0.1 N NaOH and ultrasonicated (150 W) at 55 °C for 15 min. Again, the supernatant was collected and the residue was washed three times with DW. Finally, the collected supernatant was precipitated using 95% ethanol (three-fold volume) and kept undisturbed for 12 h. The solution was then centrifuged at 8000 rpm for 20 min and the precipitate was further dried in a lyophilizer (Labconco Free Zone 6 Plus, USA) to obtain M1E (totapuri extracted soluble dietary fiber), M2E (safeda extracted soluble dietary fiber), P1E (bhagwa extracted soluble dietary fiber), and P2E (daru extracted soluble dietary fiber) (Fig. 1).

Extraction and SDF yields were calculated as shown in the equations below:

$$\text{Extraction yield(\%)} = \frac{A}{B} \times 100$$

Here, *A* signifies the sample weight after extraction and *B* signifies the initial sample weight.

$$\text{SDF yield(\%)} = \frac{A - B - C}{Y} \times 100$$

Here, *A* signifies the sample weight after extraction, *B* signifies ash content, *C* signifies protein content, and *Y* denotes the initial weight of the sample.

2.3 Chemical composition of samples

Total dietary fiber content that includes soluble dietary fiber was measured using megazyme kit (K-TDFR-200A) that follows the AOAC Method 991.43. The protocol reported by Bhatt, Kumari, Abhishek, & Gupta, 2020 had been followed for the estimation of moisture, fat, ash, and protein content [15].

2.4 Structural characterization of samples

2.4.1 SEM analysis

For SEM analysis, mounting of samples was done on the stub (aluminum stub) coated with carbon tape. The stubs were then placed inside the sputter E1010 ion sputter (Hitachi, Japan), and to make the sample conductive, it was coated

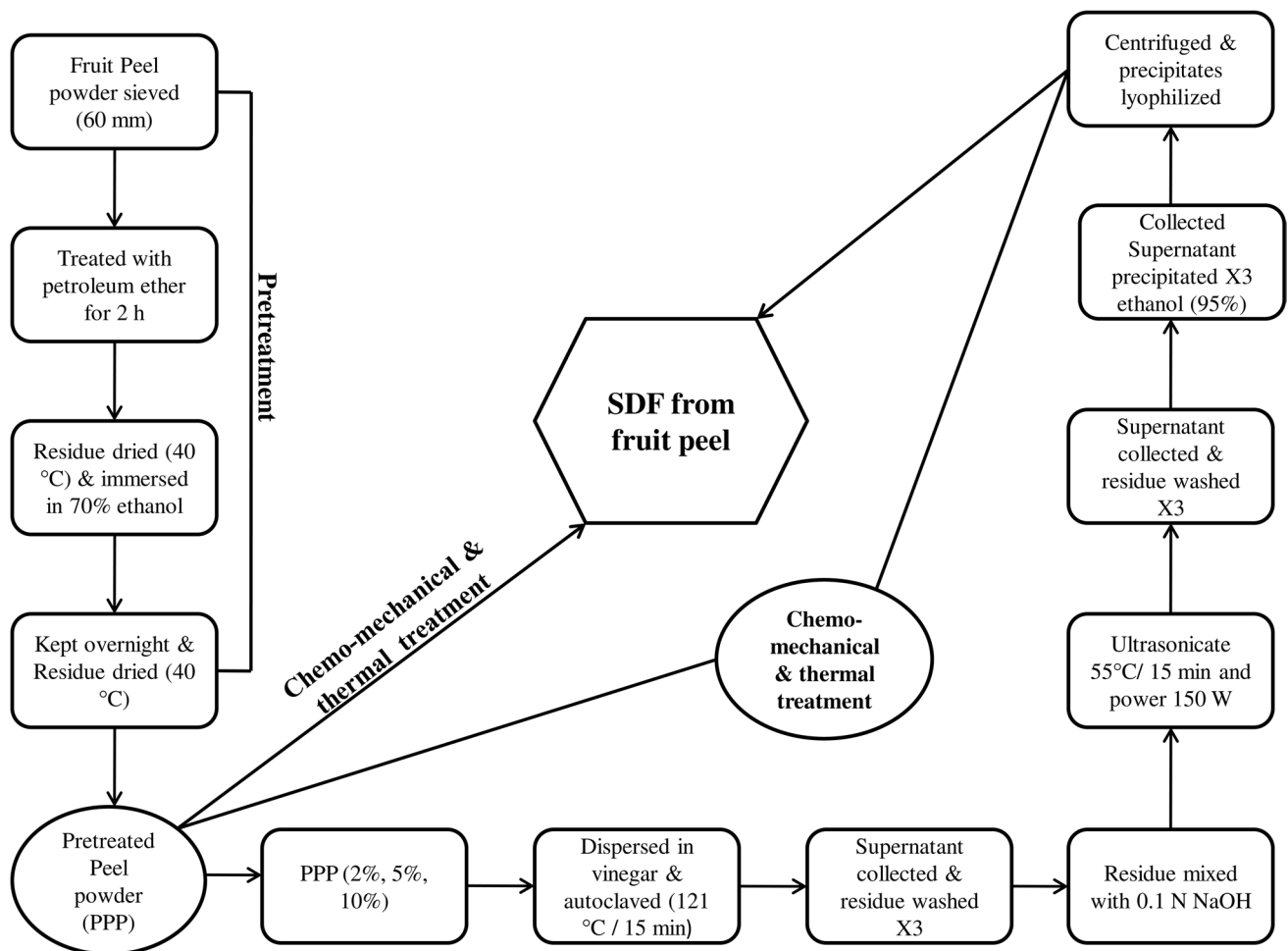


Fig. 1 Flowchart for extraction method of SDF from fruit peel

with gold for 20 s at a vacuum level of 10 Pa. Lastly, images were then captured at 15 kV on SEM (S3400N; Hitachi).

2.4.2 FTIR analysis

The PerkinElmer FT-IR spectrophotometer spectrum RX 1 (RX-1) was used for structural analysis. Each sample (10 mg) in powder form was analyzed in spectral range recorded from 4000 to 400 cm^{-1} with a resolution of 1 cm^{-1} .

2.4.3 Thermogravimetric analysis (TGA)

The thermogravimetric analysis of samples was carried out using a TGA machine. Briefly, each sample (2 mg) was analyzed under nitrogen atmosphere using a thermogravimetric analyzer (NETZSCH Geratebau GmbH STA 449 F1 Jupiter) at 10 $^{\circ}\text{C}/\text{min}$ heating rate with a temperature range of 20–400 $^{\circ}\text{C}$.

2.5 Zeta potential of samples

The charge on the surface of the sample was estimated using a particle analyzer (Zetasizer Nano ZS, Malvern Instruments Ltd). For this, sample solution (2 mg/mL) was positioned inside the capillary cell sealed via stopper and then mounted on the instrument to estimate the charge on a sample. The data represented the charge on samples quantitatively.

2.6 Viscosity measurement

The viscosity of SDF samples namely M1E, M2E, P1E, and P2E was measured using a rheometer (Anton Paar MCR 102). The SDF samples were prepared as described by Feng et al. (2017) with a measurement system having probe PP50, shear rate ranging between 0.1 and 1000 s^{-1} at temperature 25 $^{\circ}\text{C}$ [16].

2.7 Functional properties of samples

The water absorption index (WAI), oil absorption index (OAI), and water solubility index (WSI) were done as described by Dong, Wang, Hu, Long, & Lv [17]. Bulk density and water activity were carried out as described by Bhatt, Kumari, Abhishek, & Gupta [15]. Emulsion stability and emulsifying capacity were measured by the method described by Dong, Wang, Hu, Long, & Lv [17].

2.7.1 Glucose adsorption capacity (GAC)

To estimate GAC, the methodology described by Gan et al. (2020) was followed with some minor modifications [11]. As per this method, 100 mg sample was dispersed in 5 mL of glucose solution (0.5 mg/mL) and then kept in a shaking incubator for 6 h at 37 °C. Furthermore, samples were centrifuged at 4800 rpm for 10 min, and then supernatant (0.5 mL) was withdrawn with volume makeup to 3 mL using DW. To this aliquot, 3 mL of 3,5-Dinitrosalicylic acid (DNS) was added and incubated at 100 °C for 5 min with proper shaking. The absorbance was recorded at 540 nm. The GAC was estimated using the equation given below:

$$GAC(\text{mg/g}) = \frac{A1 - A2}{X1}$$

where A1 and A2 signify glucose concentration absorbed before and after the addition of fiber, and X1 represents the sample weight.

2.7.2 Sodium cholate binding capacity (SCBC)

Estimation of SCBC was carried out as described by Hu & Zhao (2018) with some modifications [13]. A total of 500 mg sample was mixed with 0.15 mol/L of NaCl solution (50 mL) having pH 7.0. To this solution, 0.5 mg/mL of sodium cholate was dispersed and then constant shaking was provided at 37 °C for 2 h. Furthermore, samples were centrifuged for 20 min at 4000 rpm and the supernatant was collected. To this supernatant (1 mL), 45% of sulfuric acid (6 mL) and 0.3% of furaldehyde (1 mL) were added and incubated for about 30 min at 65 °C. Absorbance was measured at 620 nm and SCBC was estimated as explained above.

2.7.3 Cholesterol binding capacity (CBC) and cation exchange capacity (CEC)

CBC was estimated as described by Hu & Zhao (2018) with minor modifications [13]. Egg yolk (10 mL) was mixed with DW (10 mL) and whipped gently to form an emulsion. A total of 100 mg sample was dispersed in 5 mL

of emulsion and incubated for 2 h at 37 °C with constant shaking. After incubation, the sample was centrifuged and then 1 mL supernatant was withdrawn followed by dilution to 10 times with glacial acetic acid. Furthermore, 0.2 mL supernatant was mixed with 0.8 mL of glacial acetic acid and 0.2 mL of o-phthalaldehyde in a sequence, and finally, 4 mL of an acid mixture (sulfuric and glacial acetic acid; 1:1 v/v) was added and allowed to react for 10 min. The absorbance was recorded at 550 nm, and CBC was estimated as explained above.

The method described by Yu, Bei, Zhao, Li, & Cheng (2018) was followed for the estimation of CEC with some minor modifications [18]. A total of 1 g sample was dispersed in 0.1 mol/L HCl (50 mL) and stirred for 24 h at 25 °C. The suspension was then centrifuged at 8000 rpm for 15 min and washing off the residue was carried out using DW until the supernatant pH became above 4. Thereafter, the residue was dispersed in 100 mL of sodium chloride solution (15% w/v) along with blank dispersed in water. The sample was then again centrifuged, and the supernatant was withdrawn and titrated with sodium hydroxide solution (0.1 mol/L) using phenolphthalein as an indicator. The CEC was estimated as given below:

$$CEC(\text{mmol/g}) = \frac{0.1 \times (N1 - N2)}{X1}$$

where, 0.1 is the molarity of NaOH used for titration, N1 is the titer value of NaOH for sample (mL), N2 is the titer value of NaOH for blank (mL), and X1 represents the sample weight (g).

2.8 Estimation of phenolic flavonoid and antioxidant assays

The determination of total phenolic and flavonoid contents along with anti-oxidant assays that include 2, 2'-azino-bis (3-ethylbenzothiazoline-6-sulphonic acid (ABTS) and 2, 2-diphenyl-1-picrylhydrazyl (DPPH) was estimated as described previously by Bhatt, Dadwal, Padwad, & Gupta [19].

2.9 Mineral estimation using Atomic Absorption Spectroscopy

The estimation of mineral content was carried out using atomic absorption spectrophotometer (AA-6300, Shimadzu) fitted out with cathode lamps (Single hollow) particularly for every element with an air-acetylene burner. The sample preparation was carried out using the standard association of analytical community (AOAC) wet digestion method (2010) [20].

2.9.1 Color estimation of samples

Hunter colorimeter was used for colorimetric estimation (Chroma-Meter CR-400, KONICA MINOLTA) of samples. Each sample was recorded in triplicates.

2.9.2 Statistical analysis

Each experiment was carried out in triplicates ($n = 3$), represented as mean \pm standard deviation. For ensuring higher accuracy in results, a two-way Analysis of Variance (ANOVA) was carried out with a significance level of $p < 0.05$ using Tukey's multiple comparison test.

3 Results and discussion

3.1 Extraction yield and chemical composition

The extraction yield was considered as the assessment index for the extraction of SDF at varied sample concentrations. Of these, maximum SDF yield was obtained in 5% (w/v) samples as shown in Table 1. The extraction was carried out using a combination of chemical, mechanical, and thermal methods. As per literature, the peel majorly contains pectin which gets extracted in the acidic medium [21]. A deep investigation of the literature survey also revealed a process for pectin extraction

from passion fruit via autoclaving at 121 °C for 15 min [22]. Thus, employing the use of both acidic medium and autoclaving enhanced the overall extraction yield of SDF. Contrary, alkali medium along with ultrasonic treatment has been reported for effective extraction of SDF [14]. Therefore, the maximum extraction yield was observed at 5% sample concentration as M1E ($44.35 \pm 0.66\%$), M2E ($39.87 \pm 0.27\%$), P1E ($28.76 \pm 0.12\%$), and P2E ($23.82 \pm 0.71\%$), respectively, where density, mass transfer rate, and temperature might have affected the extraction yield. It is noteworthy here that our modified method exhibited maximum extraction of SDF as compared to previous reports wherein complex enzymatic, chemical, and mechanical methods had been used [11, 13]. Thus, it is worth mentioning here that our modified extraction method is one step closer towards a greener approach that yields a good amount of SDF, and might be employed at an industrial scale due to its cheap and simple nature compared to the complex enzymatic method.

The compositional evaluation of samples with maximum yield (5% w/v) was carried out and analyzed for their purity (Table 2). The protein content of all SDF samples namely M1E, M2E, P1E, and P2E was observed to be below 1% with minimum content in M1E ($0.38 \pm 0.03\%$). The ash content was observed to be in the range of 5.24–5.35% and moisture in the range of 6.14–6.62%. The fat content of the sample was observed to be less with minimum content in P1E ($0.42 \pm 0.02\%$) which could be possibly due to pretreatment of a peel with petroleum ether increasing the overall purity of SDF.

Table 1 Extraction yield of raw and SDF samples

Sample	2% (w/v)	5% (w/v)	10% (w/v)
M1E	41.50 ± 0.40^a	45.60 ± 0.30^a	34.50 ± 0.20^a
M2E	39.30 ± 0.30^a	40.36 ± 0.25^b	35.36 ± 0.40^a
P1E	25.20 ± 0.26^b	29.40 ± 0.40^c	29.60 ± 0.20^b
P2E	23.40 ± 0.36^b	24.80 ± 0.30^d	23.50 ± 0.20^c

3.2 Structural characterization

3.2.1 SEM analysis

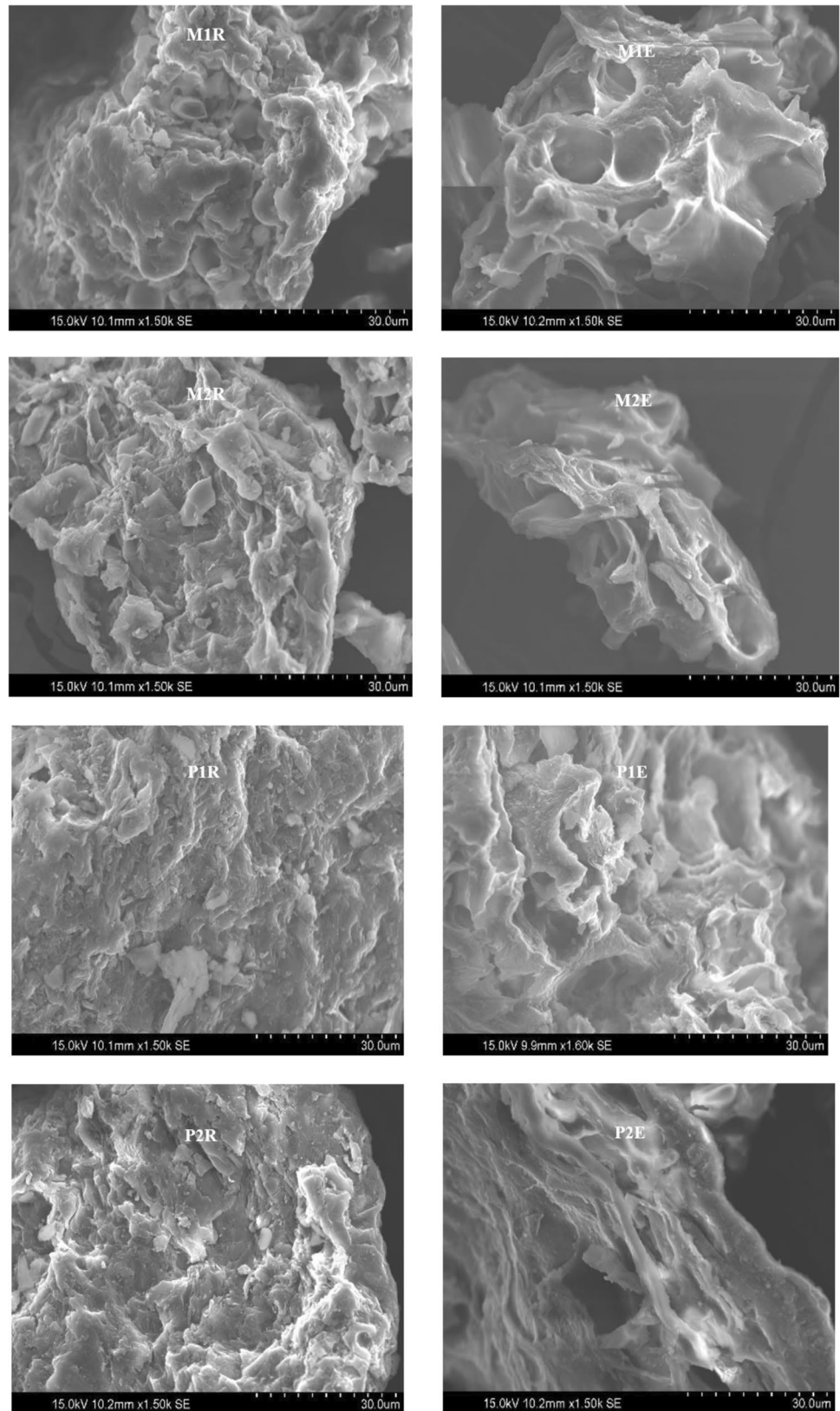
The SEM analysis revealed the microstructure of both raw and SDF samples. Interestingly, in Fig. 2, among M1R and M1E, a clear distinction in the morphology can be seen with

Table 2 Compositional analysis of raw and SDF samples

Sample	Moisture (%)	Fat (%)	Ash (%)	Protein (%)	Purity (%)	SDF (%)	TDF (%)
M1R	10.67 ± 0.01^a	4.11 ± 0.03^a	2.76 ± 0.15^a	3.75 ± 0.41^a	78.71 ± 0.15^a	14.51 ± 0.57^a	51.81 ± 0.43^a
M2R	10.44 ± 0.15^a	2.25 ± 0.18^b	4.21 ± 0.05^b	6.93 ± 0.36^b	76.17 ± 0.18^b	12.76 ± 0.38^b	46.27 ± 0.49^b
P1R	10.18 ± 0.23^{ac}	3.16 ± 0.03^c	4.10 ± 0.09^b	4.94 ± 0.38^c	77.62 ± 0.18^c	7.35 ± 0.96^c	67.39 ± 0.94^c
P2R	9.74 ± 0.21^{bc}	4.41 ± 0.06^a	2.74 ± 0.09^a	4.13 ± 0.55^a	78.98 ± 0.22^a	3.98 ± 0.98^d	59.37 ± 0.80^d
M1E	6.33 ± 0.10^b	1.18 ± 0.02^d	5.30 ± 0.15^d	0.38 ± 0.03^d	86.81 ± 0.07^d	44.35 ± 0.66^e	57.78 ± 0.07^a
M2E	6.14 ± 0.08^b	1.77 ± 0.13^b	5.32 ± 0.07^d	0.76 ± 0.07^e	86.01 ± 0.08^e	39.87 ± 0.27^f	50.09 ± 0.12^a
P1E	6.47 ± 0.10^b	0.42 ± 0.02^e	5.24 ± 0.21^d	0.41 ± 0.04^d	87.46 ± 0.09^f	28.76 ± 0.12^g	67.49 ± 0.05^c
P2E	6.62 ± 0.21^d	0.91 ± 0.03^{de}	5.35 ± 0.42^d	0.79 ± 0.07^{de}	86.33 ± 0.18^{df}	23.82 ± 0.71^h	60.74 ± 0.07^f

M1R Totapuri raw dietary fiber, M2R Safeda raw dietary fiber, P1R Bhagwa raw dietary fiber, P2R Daru raw dietary fiber, M1E Totapuri extracted soluble dietary fiber, M2E Safeda extracted soluble dietary fiber, P1E Bhagwa extracted soluble dietary fiber, P2E Daru extracted soluble dietary fiber

Fig. 2 SEM analysis of raw and SDF samples. M1R: Totapuri raw dietary fiber, M2R: Safeda raw dietary fiber, P1R: Bhagwa raw dietary fiber, P2R: Daru raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber



the raw sample as intact, rough, and rigid structure and the SDF sample as porous, and loosened structure. The alkaline treatment is known for the breakage of glycosidic linkages among the dietary fiber resulting in partial degradation of cellulose and hemicellulose leading to the porous structure of dietary fiber [23]. Apart from chemical treatment, the shear stress, turbulence, and cavitations created by the ultrasonic treatment along with variation in the temperature via autoclaving might have aided in an increment of the pores and loosening of the structure [24]. Therefore, in our study, the SEM analysis revealed the effect of modified chemo-mechanical and thermal treatment in improving the overall microstructure of fiber with a positive effect on its physico-chemical properties.

3.2.2 FT-IR analysis

The FT-IR spectrum is known to explicate the presence of functional groups along with their bonding information that elucidates the structural characteristics of dietary fiber. In the current study, the FT-IR analysis was carried out to investigate the effect of modified extraction treatment on SDF as shown in Fig. 3(a). The characteristic band ranging from 3411.8 to 3433.8 cm^{-1} exhibited a similar trend in shifting to higher wave-number among all SDF samples namely M1E, M2E, P1E, and P2E when compared to their respective raw samples namely M1R, M2R, P1R, and P2R. This up-shift in characteristic peak could be ascribed to the stretching of the $-\text{OH}$ bond which in turn weakens the hydrogen bond majorly among cellulose and hemicellulose polysaccharides. Interestingly, these results exhibit the partial degradation of cellulose or hemicellulose due to strong extraction conditions increasing the overall SDF yield. The peak ranging from 2922.1 to 2928.1 cm^{-1} corresponds to stretching of the $-\text{C}-\text{H}$ group that represents characteristics of typical polymer along with $-\text{H}-\text{C}-\text{H}$ and $-\text{C}-\text{O}-\text{H}$ conjugated bending vibrations [23]. A close inspection of spectral data reflects the decrease in intensity of this particular peak among all SDF samples signifying the breakage of $-\text{C}-\text{H}$ and hydrogen bonds within dietary fiber due to alkali treatment. The breakage in $-\text{C}-\text{H}$ and hydrogen bond among polysaccharides majorly cellulose and hemicellulose would thereby help in loosening of fiber structure increasing its overall functional properties [23]. The peaks ranging from 1736.7 to 1746.9 cm^{-1} represent the stretching of $\text{C}=\text{O}$ carbonyl groups among hemicellulose, pectin, or lignin. The up-shift in this particular peak among all SDF samples namely M1E, M2E, P1E, and P2E along with the complete disappearance in the P1E sample could be attributed to the alkali degradation of complex polysaccharides increasing the overall SDF content. The bands ranging from 1618.6 to 1623.8 cm^{-1} signify the stretching or bending of lignin hydrocarbons along with the presence of uronic acid. The higher peak intensity

signifies the presence of a higher number of uronic acid moieties in all SDF samples when compared to their respective raw samples namely M1R, M2R, P1R, and P2R which might have resulted in degradation of lignin. The down-shift in peak from 1419.7 to 1445.8 cm^{-1} signifies the weakening of $\text{C}-\text{H}$ groups either aliphatic or aromatic, originating probably from lignin indicating lignin degradation [17, 23]. On further inspecting the spectral data, a great reduction along with shifting and complete disappearance of characteristic peaks was observed in the range from 1700 to 1200 cm^{-1} among all SDF samples compared to their respective raw samples namely M1R, M2R, P1R, and P2R. This observation indicates the partial degradation of cellulose, hemicellulose, pectin, lignin, and protein content thus increasing the overall SDF yield along with loosening of intact fiber structure. Finally, the peak intensity ranging from 1000 to 1150 cm^{-1} signifies the stretching vibration of $-\text{C}-\text{O}$ bonds, probably due to combined vibrations from primary alcohol groups or $\text{C}-\text{O}-\text{C}$ and $\text{C}-\text{O}-\text{H}$ groups of the sugar rings [11]. Therefore, this critical observation explicitly signifies the effect of modified extraction conditions on the structure and overall yield of all SDF samples.

3.2.3 Thermogravimetric analysis

The thermal disintegration of all raw (M1R, M2R, P1R, and P2R) and SDF samples (M1E, M2E, P1E, and P2E) was carried out via thermogravimetric analyzer (Fig. 3b). The TGA was carried out within the temperature of 50–400 °C exhibiting changes in weight loss and degradation characteristics of the samples. The thermal degradation could be majorly divided into three main stages 50–150 °C, 150–250 °C, and 250–400 °C [25].

The first stage ranged from 50 to 150 °C, where a major loss in weight was observed due to evaporation of absorbed water, degradation of various polysaccharides of low molecular weight, and devolatilization that majorly occurs at 121 °C [23, 25].

The second stage reflects the weight loss ranging from 150 to 250 °C. The onset temperature where an abrupt change in degradation characteristics was observed ranged from 158.36 to 186.28 °C. A weight loss of up to 20% was observed in this stage with degradation temperatures for M1R, M2R, P1R, P2R, M1E, M2E, P1E, and P2E as 210.39 °C, 215.91 °C, 236.95 °C, 258.88 °C, 215.50 °C, 214.32 °C, 208.62 °C, and 231.48 °C, respectively. This reduction in weight loss could be attributed to the disintegration of fiber by dihydroxylation, decarboxylation, or deoxygenation [2]. The results are as per previously reported literature where the 20% degradation of soluble dietary fiber from coffee peel was observed in the temperature range of 130–250 °C [17].

The third stage (250–400 °C) corresponds to major weight loss in all samples, i.e., up to 60% (Supplementary Table 1). The loss in mass was abrupt at this stage with final degradation

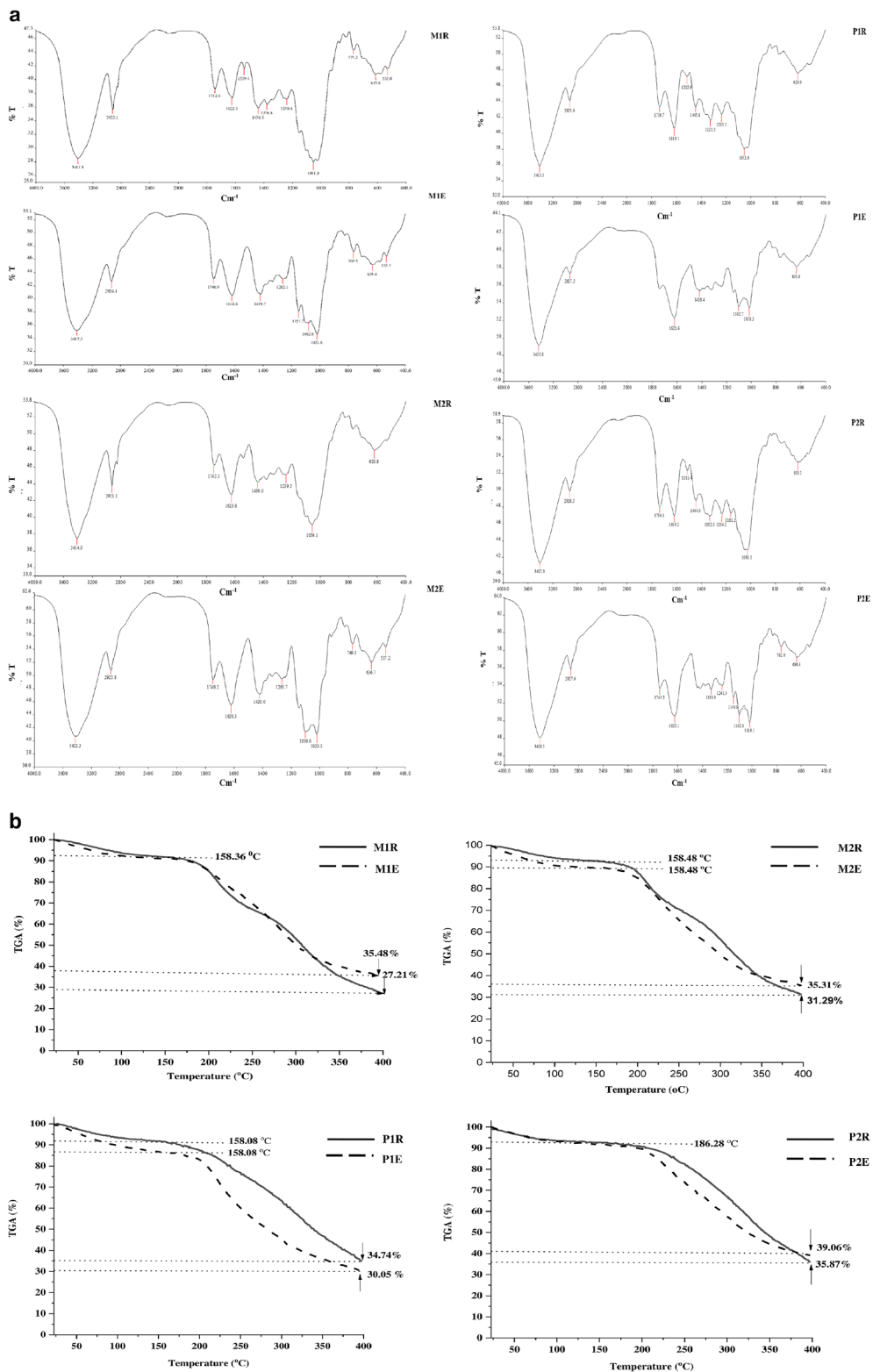


Fig. 3 (a): FT-IR spectra of raw and SDF samples. (b): TGA analysis of raw and SDF samples. MIR: Totapuri raw dietary fiber, M2R: Safeda raw dietary fiber, P1R: Bhagwa raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber

raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber

temperatures for M1R, M2R, P1R, P2R, M1E, M2E, P1E, and P2E as 333.39 °C, 343.91 °C, 376.95 °C, 382.88 °C, 345.50 °C, 345.32 °C, 322.62 °C, and 388.48 °C, respectively. This rapid reduction in mass could be attributed to the thermal degradation of hemicelluloses, pectin, and lignin [25]. Pyrolytic degradation of polysaccharides mainly pectin and hemicelluloses is majorly responsible for this mass reduction [23]. The weight loss at higher temperatures has also been related to the molecular weight of samples exhibiting a positive correlation [25]. Moreover, the residual mass left at 398.62 °C for samples namely M1R, M2R, P1R, P2R, M1E, M2E, P1E, and P2E is 27.21%, 31.29%, 34.74%, 35.87%, 35.48%, 35.31%, 30.05%, and 39.06%, respectively (Supplementary Table 1). The residual mass of all treated samples was observed to be more if compared to their respective raw samples namely M1R, M2R, P1R, and P2R which signifies the enhanced thermal stability of SDF samples. As per literature, the temperature range from 400 to 600 °C has been reported for weight loss majorly due to lignin and various other complex polysaccharides [25]. Therefore, the results from TGA analysis indicate the positive effect of the extraction mechanism on the thermal stability of all SDF samples compared to their respective raw samples, thereby enhancing its application in various food product formulations.

3.3 Zeta potential

The zeta potential indicates the charge on the surface of a material which is directly related to its gelation capability. The presence of a greater negative charge on the surface of SDF indicates its ability to merge with cation along with higher dissociation producing strong electrostatic attraction [11, 26]. In the present study, all raw samples namely M1R, M2R, P1R, and P2R exhibited -8.59 , -14.96 , -20.43 , and -18.6 mV zeta potential, respectively. In contrast, the SDF samples namely M1E, M2E, P1E, and P2E exhibited zeta potential as -16.43 , -18.96 , -22.93 , and -20.66 mV which suggested an increase in surface charge as 91.2%, 26.7%, 12.2%, and 11.1%, respectively, with the maximum increase in M1E sample. A greater negative charge creates a strong electrostatic force and helps in extending the molecular chain completely. This helps to create intermolecular crosslinking which aids in the formation of stronger gels. Thus, the surface charge strongly affects the stability and dispersion capability of SDF along with its rheological behavior in an aqueous solution.

3.4 Viscosity measurements

Viscosity is considered as one of the crucial physical parameters of SDF that enables a complete understanding of changing conformations and their application. The flow behavior of SDF samples namely M1E, M2E, P1E, and P2E at various

concentrations is shown in Fig. 4 which illustrates the effect on viscosity with change in shear strain. In general, the viscosity increased with an increase in SDF concentration with maximum viscosity in all 6% (w/v) formulations for M1E, M2E, P1E, and P2E samples. The data illustrated the pseudoplastic behavior of all SDF samples that exhibited the shear thinning behavior with respect to shear strain. However, with an increase in shear strain, the samples exhibited Newtonian fluid behavior as they showed no change with an increase in shear strain. Though, a reduction in the number of entanglements among chains could also lead to reduced viscosity at higher shear rates. Thereby, our modified SDF samples can act as a better alternative for a thickening agent in food industries.

3.5 Functional properties of samples

The WAI signifies the association of water depending on the chemical structure, stability, density, nature, amount, and site of attachment (hydrophilic) of dietary fiber. In the present study, the water absorption index of both SDF samples (M1E, M2E, P1E, and P2E) and raw samples (M1R, M2R, P1R, and P2R) ranged from 5.54 to 10.85 g/g (Table 3). This significant increase ($p < 0.05$) in the absorption capacity among all SDF samples could be attributed to the rupturing of hydrogen bond interaction in hemicellulose rendering more hydrophilic groups for maximum interaction and increased absorption [23]. Interestingly, our results exhibited efficient WAI with maximum content in the P2E sample as 10.85 ± 0.24 g/g which is higher than SDF extracted from papaya and orange peel [14, 27]. Similarly, the WSI of SDF samples was observed to be significantly higher ($p < 0.05$) than their respective raw samples namely M1R, M2R, P1R, and P2R with maximum capacity in the M1E sample as $56.00 \pm 0.02\%$ (Table 3). This increase is probably due to three-dimensional structural change in SDF with a simultaneous increase in short-chain fiber.

The OAI depends on the surface property, hydrophobicity, and charge density of hydrocolloids. In the present study, an increase in OAI was observed in SDF samples namely M1E, M2E, P1E, and P2E as compared to their respective raw samples namely M1R, M2R, P1R, and P2R with maximum absorption in the M2E sample as 7.91 ± 0.24 g/g (Table 3). This increase among SDF samples could be observed due to chemo-mechanical and thermal treatment that might have exposed functional groups via degradation of IDF for maximum oil entrapment [10]. The materials with high bulk density generally have a low surface area and low lipid-binding capacity. Interestingly, our results exhibit a similar trend with all SDF samples showing higher OAI and low bulk density (1.70 ± 0.07 g/cm³) as shown in Table 3. The water activity of the sample is positively correlated to its moisture content. The water activity of SDF samples namely M1E, M2E, P1E, and P2E was found low when compared to raw

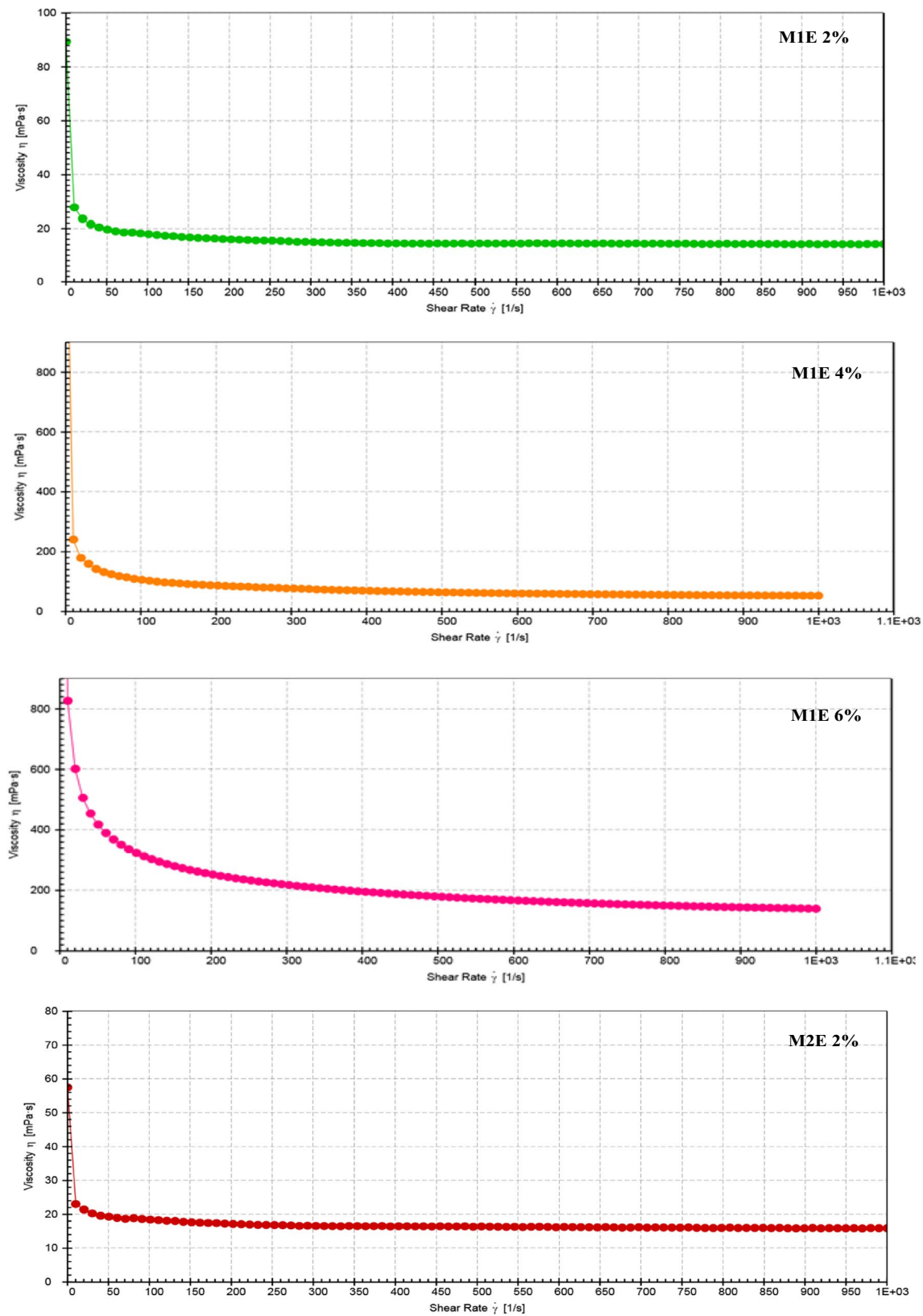


Fig. 4 Viscosity of M1E, M2E, P1E, and P2E samples. M1R: Totapuri raw dietary fiber, M2R: Safeda raw dietary fiber, P1R: Bhagwa raw dietary fiber, P2R: Daru raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary

fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber. Where, 2%, 4%, and 6% w/v denote the sample to solvent ratio

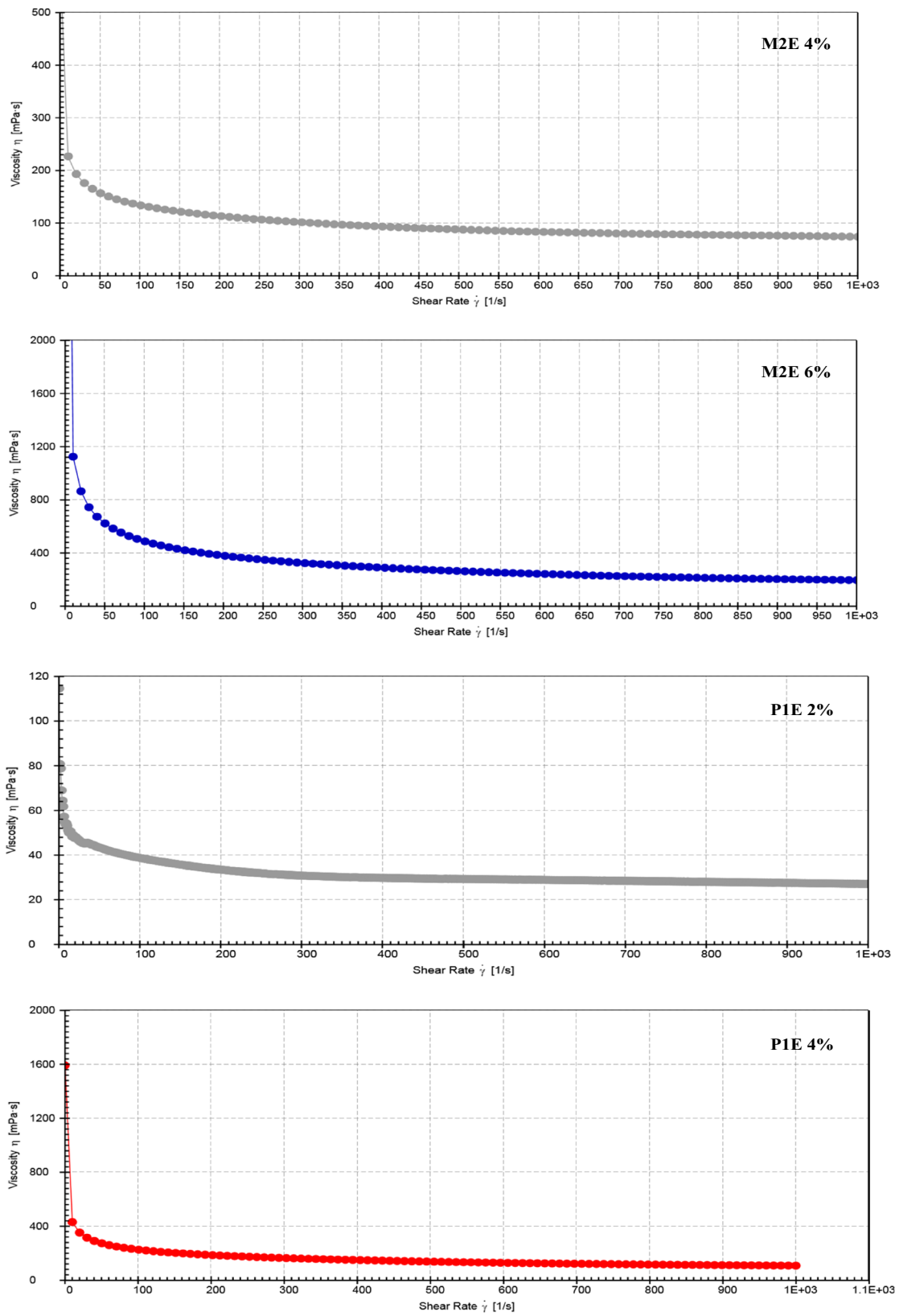


Fig. 4 (continued)

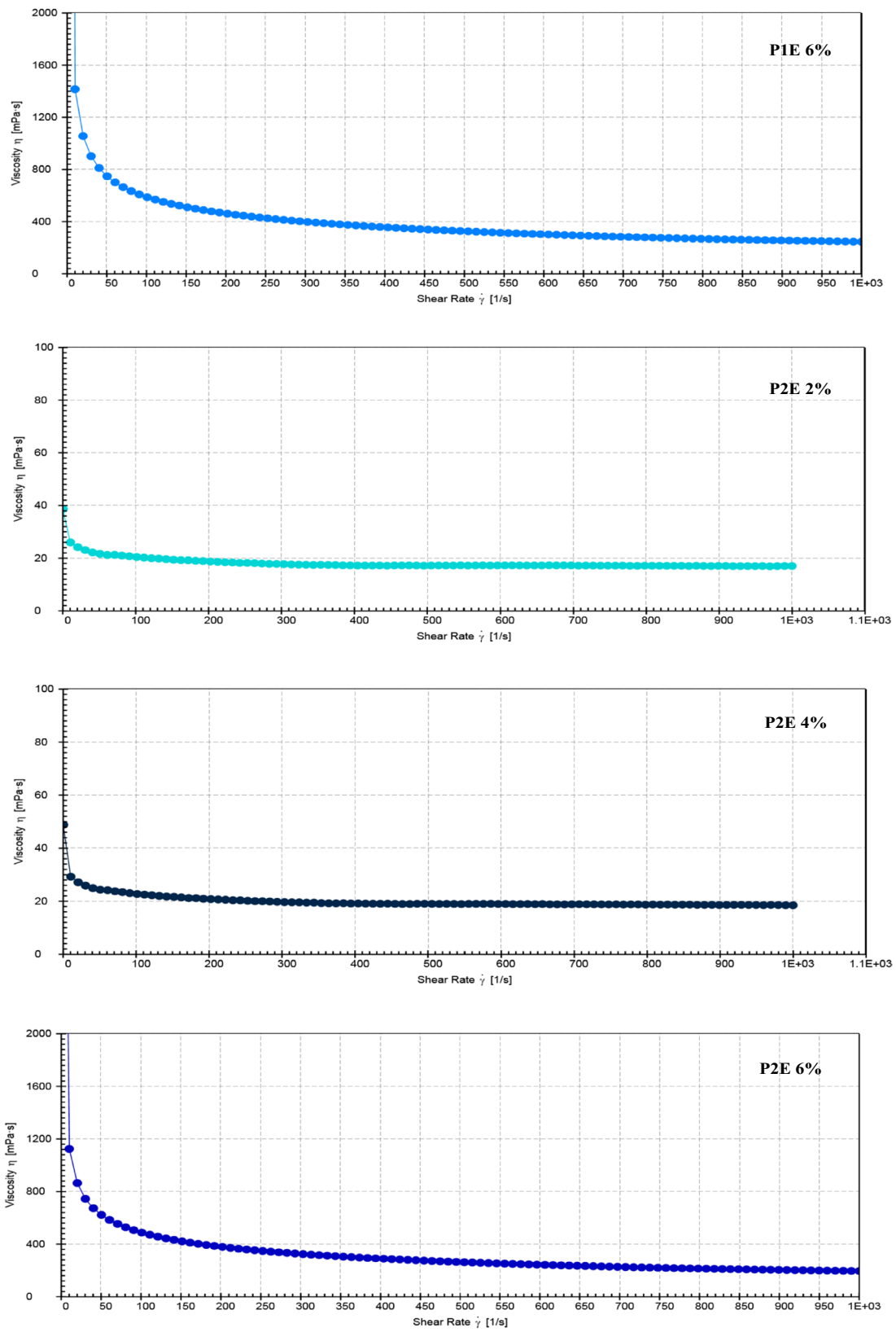


Fig. 4 (continued)

Table 3 Functional composition of raw and SDF samples

Sample	WSI (%)	WAI (g g ⁻¹)	OAI (g g ⁻¹)	Water activity (a _w)	Bulk density (g cm ⁻³)	EC (%)	ES (%)
M1R	33.96 ± 0.01 ^a	4.54 ± 0.37 ^a	2.73 ± 0.24 ^a	0.39 ± 0.07 ^a	8.72 ± 0.13 ^a	43.33 ± 0.03 ^a	15.50 ± 0.09 ^a
M2R	39.92 ± 0.09 ^b	5.15 ± 0.38 ^a	3.00 ± 0.25 ^{a,b}	0.46 ± 0.04 ^a	5.92 ± 0.02 ^a	44.16 ± 0.01 ^a	19.16 ± 0.02 ^{a,b}
P1R	21.22 ± 0.01 ^a	4.25 ± 0.25 ^a	2.20 ± 0.24 ^a	0.42 ± 0.04 ^a	8.34 ± 0.02 ^a	32.50 ± 0.02 ^b	27.50 ± 0.05 ^b
P2R	17.64 ± 0.01 ^a	5.38 ± 0.24 ^a	2.33 ± 0.14 ^{a,b}	0.53 ± 0.04 ^a	5.26 ± 0.12 ^a	37.50 ± 0.02 ^a	23.50 ± 0.05 ^{b,c}
M1E	56.00 ± 0.02 ^b	7.25 ± 0.25 ^a	6.33 ± 0.14 ^{a,b}	0.18 ± 0.03 ^a	2.84 ± 0.08 ^a	73.33 ± 0.02 ^c	42.50 ± 0.08 ^d
M2E	53.89 ± 0.01 ^b	10.22 ± 0.24 ^b	7.91 ± 0.24 ^b	0.14 ± 0.01 ^a	1.70 ± 0.07 ^a	77.50 ± 0.02 ^c	47.50 ± 0.05 ^d
P1E	38.66 ± 0.03 ^a	10.83 ± 0.28 ^b	4.71 ± 0.37 ^{a,b}	0.16 ± 0.01 ^a	4.00 ± 0.07 ^a	77.48 ± 0.06 ^d	45.00 ± 0.05 ^d
P2E	40.50 ± 0.03 ^a	10.85 ± 0.24 ^a	5.87 ± 0.24 ^a	0.13 ± 0.04 ^a	3.97 ± 0.09 ^a	68.33 ± 0.07 ^c	44.16 ± 0.02 ^d

M1R Totapuri raw dietary fiber, M2R Safeda raw dietary fiber, P1R Bhagwa raw dietary fiber, P2R Daru raw dietary fiber, M1E Totapuri extracted soluble dietary fiber, M2E Safeda extracted soluble dietary fiber, P1E Bhagwa extracted soluble dietary fiber, P2E Daru extracted soluble dietary fiber. WSI water solubility index, WAI water absorption index, OAI oil absorption index, EC emulsion capacity, ES emulsion stability

samples namely M1R, M2R, P1R, and P2R (Table 3). The low water activity of all SDF samples exhibited their stability along with less susceptibility to microbial degradation.

The EC signifies the ability of an agent to facilitate the dispersion or solubilization of any two immiscible liquids, while ES is the capacity to sustain emulsion with resistance to rupture. In Table 3, treated samples exhibited a significant ($p < 0.05$) increase with maximum EC and ES as 77.50 ± 0.02 mL/100 mL and 47.50 ± 0.05 mL/100 mL, respectively, in the M2E sample. It is noteworthy here that the EC of the present study was higher as compared to potato pectin previously reported in the literature [28]. The EC of SDF signifies the ability of an agent for adsorption of biliar acids having efficient health effects in feces excretion, and limiting their absorption consequently reducing cholesterol levels [27].

3.5.1 GAC, SCBC, and CBC analysis

The ability of DF to exhibit viscosity and gel-forming ability helps in reducing the abdominal emptying along with adsorption of micronutrients from the gut which helps in postprandial glucose reduction [13]. In addition, DF is known to reduce cholesterol and fat content [13]. As depicted in Fig. 5, a dietary fiber extracted using a modified method namely M1E, M2E, P1E, and P2E exhibited higher GAC, SCBC, and CBC compared to the raw samples. The results signified that the modified extraction method might have made the structure more porous and loosened exposing all polar and non-polar groups rendering interaction between molecules and DF exhibiting potential health benefits.

3.5.2 CEC analysis

The effect of modified chemo-mechanical and thermal treatment on CEC exhibited a significant increase ($p < 0.05$) among SDF samples namely M1E, M2E, P1E, and P2E

compared to the raw samples namely M1R, M2R, P1R, and P2R (Fig. 5). This increase could be probably due to a decrease in lignin content. Lignin is known to contribute to non-covalent electrostatic interactions due to the presence of various phenolic and carboxylic surface groups that tend to decrease the hydrophobicity. The FT-IR results of the present study confirmed the decrease in lignin content, with an increase in uronic acid content along with interruption in cellulose and hemicellulose linkages. The principal functional groups in DF that are known to influence the CEC include hydroxyl and carboxyl phenols where cations might replace H⁺ ions thereby enhancing metal chelation and increasing overall CEC [25]. Therefore, the present study highlights the significant increase in CEC among all SDF samples.

3.6 TPC, TFC, and antioxidant activity

As shown in Table 4, the TPC and TFC content in all SDF samples namely M1E, M2E, P1E, and P2E exhibited a significant decrease ($p < 0.05$) compared to the raw samples namely M1R, M2R, P1R, and P2R. This reduction could be possibly due to the leaching of phenolic and flavonoid compounds in the extraction medium. Furthermore, a longer duration of ethanolic precipitation along with filtration has been reported for the reduction of phenolic compounds [18]. Therefore, the chemo-mechanical and thermal treatment might have resulted in maximum phenolic extraction in the surrounding medium with few phenolic and flavonoid molecules bound to SDF.

Numerous methods have been employed for the estimation of antioxidant activity. Therefore, in the present study, DPPH and ABTS activities were performed to estimate the antioxidant potential. The results exhibited a significant decrease in the activity of SDF samples namely M1E, M2E, P1E, and P2E compared to raw samples namely M1R, M2R, P1R, and P2R (Table 4) observed during TPC

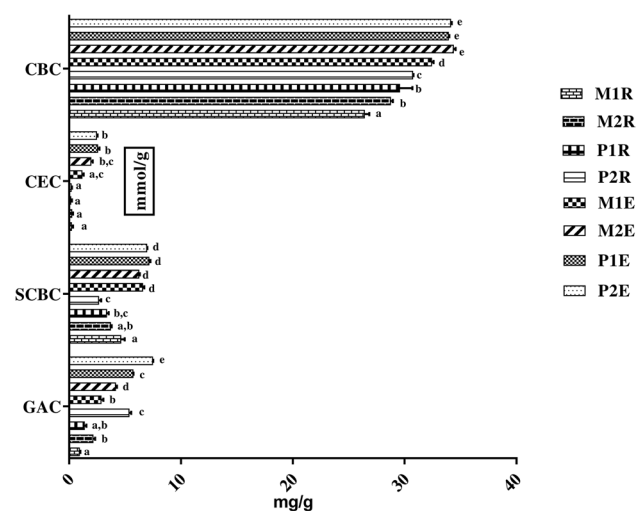


Fig. 5 GAC, SCBC, CEC, and CBC of raw and SDF samples. MIR: Totapuri raw dietary fiber, M2R: Safeda raw dietary fiber, P1R: Bhagwa raw dietary fiber, P2R: Daru raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber

and TFC estimation demonstrating a positive correlation between them [18]. It is noteworthy here that even after pretreatment and harsh chemo-mechanical treatment, substantial amount of TPC and TFC with maximum content in P2E sample $105.04 \mu\text{g GAE/mg}$ and $80.58 \mu\text{g RU/mg}$, respectively, was observed compared to other SDF samples which signify its potential as fiber bound phenolics and flavonoids. Therefore, the treated samples exhibited efficient TPC, TFC, and anti-oxidant activity that might be used in food formulations.

3.7 Mineral estimation using AAS

Minerals are regarded as one of the essential components for maintaining a balanced physiological response. In the present study, 7 minerals that include both major and minor micronutrients were detected in both raw and SDF samples. The probable reason for this could be the change in the ionic concentration caused by the reagents used during the process of extraction [29]. Secondly, the autoclaving treatment along with ultrasonication is known for leaching of minerals in the surrounding media making them more available for binding with SDF [30]. Thirdly, the charged polysaccharides are known for binding up minerals with them especially charged pectin [31]. Finally, the autoclaving treatment tends to reduce the anti-nutritional components majorly phytic acid, increasing the overall mineral content as phytic acid possesses the mineral chelating property; thereby, reduction in phytic acid will tend to release the bound minerals and hence increasing overall mineral content in SDF samples

[32]. The mineral content of all samples exhibited significant differences ($p < 0.05$) as shown in Table 5. Thereby, the incorporation of SDF in various food products can act as a mineral fortification.

3.8 Color analysis of samples

The color analysis of raw and SDF samples was measured as the ' L^* ', ' a^* ', and ' b^* ' value with ΔE as the color difference. In Fig. 6, the ' L^* ' values of raw samples namely M1R, M2R, P1R, and P2R were higher than SDF samples namely M1E, M2E, P1E, and P2E which signified that SDF samples were darker probably due to alkali extraction which is as per previously reported literature [33]. The negative decrease in the ' a^* ' value in SDF samples of mango namely M1E and M2E was observed compared to the respective raw samples namely M1R and M2R which signifies the greenness of samples. Conversely, in the case of pomegranate, a positive increasing trend was observed in SDF samples compared to their respective raw samples. This indicated the significant increase in redness of SDF samples namely P1E and P2E when compared to raw samples namely P1R and P2R imparting overall darkness to SDF samples. Finally, a positive decrease in the ' b^* ' value in all SDF samples was observed compared to their respective raw samples. This indicated the decreasing yellowness in all SDF samples that could be probably due to the presence of some impurities namely starch or denatured protein [34]. The color value of dietary fiber plays a vital role in its application in the food industry. Thus, the present study revealed that SDF can

Table 4 Phenolic, flavonoid, and antioxidant activities (DPPH & ABTS) of raw and SDF samples

Samples	Total phenolic content ($\mu\text{g GAE/mg}$)	Total flavonoid content ($\mu\text{g RU/mg}$)	IC ₅₀ of free radical scavenging activities ($\mu\text{g/mL}$)	
			DPPH	ABTS
M1R	59.46 ± 0.04^a	39.75 ± 0.04^a	15.27	11.76
M2R	$64.25 \pm 0.02^{a,c}$	27.82 ± 0.03^a	13.84	10.01
P1R	605.85 ± 0.02^b	204.58 ± 0.06^a	3.88	0.53
P2R	718.56 ± 0.03^b	212.23 ± 0.05^a	2.34	1.22
M1E	$12.17 \pm 0.08^{a,e}$	9.17 ± 0.07^a	303.96	157.54
M2E	14.16 ± 0.01^a	4.83 ± 0.03^a	282.96	282.20
P1E	91.48 ± 0.01^a	75.81 ± 0.03^a	105.29	70.43
P2E	105.04 ± 0.01^a	80.58 ± 0.03^a	104.75	73.56
Ascorbic acid	-	-	1.05	2.01

M1R Totapuri raw dietary fiber, M2R Safeda raw dietary fiber, P1R Bhagwa raw dietary fiber, P2R Daru raw dietary fiber, M1E Totapuri extracted soluble dietary fiber, M2E Safeda extracted soluble dietary fiber, P1E Bhagwa extracted soluble dietary fiber, P2E Daru extracted soluble dietary fiber

Table 5 Mineral composition of raw and SDF samples

Sample	Ca (mg/100 g)	Mg (mg/100 g)	Na (mg/100 g)	Fe (mg/100 g)	Zn (mg/100 g)	Cu (mg/100 g)	Mn (mg/100 g)
M1R	14.99 ± 0.006 ^a	25.89 ± 0.029 ^a	5.79 ± 0.003 ^a	6.08 ± 0.002 ^a	0.89 ± 0.001 ^a	0.48 ± 0.003 ^a	0.65 ± 0.003 ^a
M2R	11.74 ± 0.004 ^b	29.30 ± 0.023 ^{a,d}	8.39 ± 0.007 ^b	10.08 ± 0.004 ^b	2.40 ± 0.001 ^b	0.68 ± 0.007 ^b	0.86 ± 0.004 ^a
P1R	53.94 ± 0.018 ^c	29.56 ± 0.025 ^{a,d}	18.54 ± 0.046 ^c	24.83 ± 0.003 ^c	3.98 ± 0.004 ^c	0.70 ± 0.007 ^b	0.72 ± 0.005 ^a
P2R	59.25 ± 0.009 ^d	38.43 ± 0.012 ^a	11.64 ± 0.010 ^b	21.85 ± 0.003 ^d	4.86 ± 0.001 ^c	0.60 ± 0.002 ^a	0.31 ± 0.006 ^b
M1E	56.14 ± 0.005 ^e	57.81 ± 0.005 ^b	74.01 ± 0.078 ^d	3.50 ± 0.006 ^e	1.94 ± 0.002 ^a	0.18 ± 0.001 ^a	0.70 ± 0.005 ^b
M2E	52.51 ± 0.005 ^f	51.02 ± 0.021 ^{a,d}	76.71 ± 0.072 ^{d,e}	9.03 ± 0.001 ^f	3.94 ± 0.007 ^b	0.31 ± 0.002 ^a	0.97 ± 0.003 ^b
P1E	94.87 ± 0.017 ^g	42.89 ± 0.009 ^{a,d}	93.77 ± 0.014 ^e	22.78 ± 0.001 ^g	4.66 ± 0.002 ^{b,c}	0.22 ± 0.003 ^a	0.80 ± 0.005 ^b
P2E	100.81 ± 0.018 ^h	40.76 ± 0.009 ^c	90.50 ± 0.073 ^e	20.10 ± 0.001 ^h	4.16 ± 0.001 ^c	0.57 ± 0.007 ^b	0.40 ± 0.003 ^b

M1R Totapuri raw dietary fiber, M2R Safeda raw dietary fiber, P1R Bhagwa raw dietary fiber, P2R Daru raw dietary fiber, M1E Totapuri extracted soluble dietary fiber, M2E Safeda extracted soluble dietary fiber, P1E Bhagwa extracted soluble dietary fiber, P2E Daru extracted soluble dietary fiber

be easily incorporated into various food products increasing its overall nutritional value.

4 Conclusion

In the present study, we investigated the effect of the modified extraction method on yield, structural, chemical, and other characteristic properties of SDF. It was observed that the modified extraction method resulted in high SDF yield with maximum content in M1E samples with enhanced physicochemical and structural properties when compared to raw samples. In view of various parameters such as extraction yield, purity, enhanced

physicochemical properties, and industrial applicability our modified method substantiated all constraints. Thereby, the study aims to valorize waste to a value-added substance along with finding a novel extraction method with efficient yield and possible feasibility at an industrial scale.

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Declarations

Conflict of interest The authors declare no competing interests.

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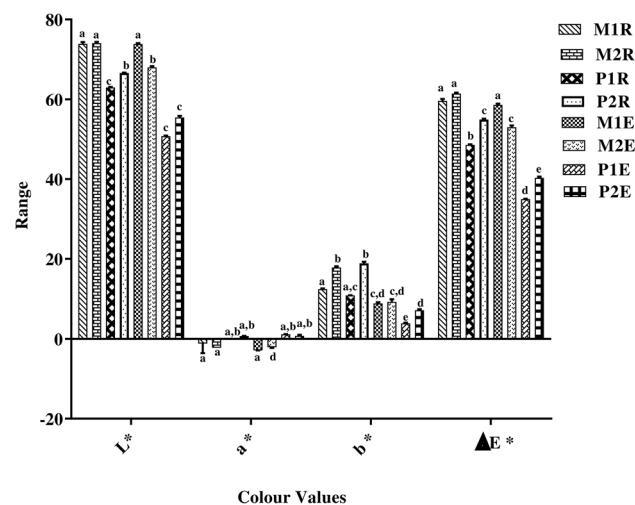


Fig. 6 Color analysis of raw and SDF samples. M1R: Totapuri raw dietary fiber, M2R: Safeda raw dietary fiber, P1R: Bhagwa raw dietary fiber, P2R: Daru raw dietary fiber, M1E: Totapuri extracted soluble dietary fiber, M2E: Safeda extracted soluble dietary fiber, P1E: Bhagwa extracted soluble dietary fiber, and P2E: Daru extracted soluble dietary fiber

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